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SATURATED DIFFUSION CELL EXPERIMENT

LOS ALAMOS
QUALITY PROGRAM

APPROVAL FOR RELEASE	
S. C. WEAVER - PREPARER Signature on file	DATE Date on file
I. R. TRIAY - PRINCIPAL INVESTIGATOR Signature on file	DATE Date on file
M. C. CLEVENGER - QUALITY ASSURANCE PROJECT LEADER Signature on file	DATE Date on file

Los Alamos
Yucca Mountain Site
Characterization Project

HISTORY OF REVISION

REVISION NO.	EFFECTIVE DATE	PAGES REVISED	REASON FOR CHANGE
R0	04/30/89	N/A	Initial procedure.
R1	03/25/92	All	Complete rewrite into new format; procedural steps slightly modified throughout.
R2	06/10/94	All	Complete rewrite into new format per LANL-YMP-QP-06.3, R1. This procedure was formerly identified as LANL-INC-DP-66.
R3	12/23/96	All	Revised to comply with LANL-YMP-QP-06.3 requirements.

SATURATED DIFFUSION CELL EXPERIMENT

1.0 PURPOSE

The purpose of this procedure is to study the rate of movement of radionuclides through tuff by contacting rock wafers with a solution containing the radionuclide of interest.

2.0 SCOPE

This procedure applies to the fabrication, assembly, preparation, and sampling of saturated diffusion cell experiments within the Dynamic Transport Task of the Los Alamos National Laboratory (LANL) Yucca Mountain Site Characterization Project (YMP).

3.0 REFERENCES

LANL-YMP-QP-02.7, Personnel Training
LANL-YMP-QP-03.5, Documenting Scientific Investigations
LANL-YMP-QP-08.1, Identification and Control of Samples
LANL-YMP-QP-12.3, Control of Measuring and Test Equipment and Standards
LANL-YMP-QP-17.6, Records Management
LANL-INC-DP-35, pH Measurement
LANL-INC-DP-60, Preparation of NTS Samples for LANL YMP Solid Core Experiments

4.0 DEFINITIONS

4.1 Planimeter

Instrument for measuring the area of an irregular flat surface.

4.2 Tracer Solution

The tracer solution is the solution containing the radionuclide(s) that will be studies.

5.0 RESPONSIBILITIES

The following personnel are responsible for the activities identified in Section 6.0 of this procedure:

- Principle Investigator (PI)
- Users of this Detailed Procedure (DP)

6.0 PROCEDURE

The use of this procedure must be controlled as follows:

- If this procedure cannot be implemented as written, YMP personnel should notify appropriate supervision. If it is determined that a portion of the work cannot be accomplished as described in this QP, or would result in an undesirable situation, that portion of the work will be stopped and not resumed until this procedure is modified, replaced by a new document, or the current work practice is documented in accordance with QP-03.5, Section 6.1.6.

- Employees may use copies of this procedure printed from the controlled document electronic file; however, employees are responsible for assuring that the correct revision of this procedure is used.

- When this procedure becomes obsolete or superseded, it must be destroyed or marked “superseded” to ensure that this document is not used to perform work.

6.1 Principle

This procedure allows the study of the transport of radioisotopic tracers through a saturated diffusion cell under static conditions. A solution containing a tracer is placed in contact with one side of a rock and periodic samples are taken of solution on the other side to determine the rate of diffusion of the tracer through the Yucca Mountain tuffs.

6.2 Equipment and Hardware/Software

The following equipment, or its equivalent, is used in this procedure:

- diamond slab saw
- lathe
- mill
- drill press
- planimeter
- vacuum oven
- constant temperature water bath unit

6.2.1 Equipment Malfunctions

During the process of this experiment, there are no critical equipment malfunctions that are likely to occur that would interrupt the experiment.

6.2.2 Safety Considerations

Ensure compliance with CST Division Environmental Safety and Health Operational Policy Statement.

6.2.3 Special Handling

There are no special handling requirements.

6.3 Preparatory Verification

If the equipment needed to conduct this experiment is found to be unclean or damaged, do not start the experiment until the problem is corrected (i.e., equipment is cleaned, repaired, or new equipment is procured). A visual inspection of each specimen within the diffusion cell shall be done. If a crack or chip is detected, the specimen should not be used.

6.3.1 Hold Points

There are no hold points for this procedure.

6.3.2 Calibration

6.3.2.1 Balances used for weighing are required to be calibrated pursuant to LANL-YMP-QP-12.3. When data are collected from a balance, the unique identifier number of that balance is recorded in the user's laboratory notebook along with the data collected.

6.3.2.2 The pH meter used for any pH determinations shall be used in accordance with DP-35. The unique identifier of the pH meter shall be recorded in the user's laboratory notebook.

6.3.2.3 The planimeter used in this experiment is an instrument that does not require calibration. Its operation is checked by measuring the area of a geometric figure of known area, calculating any necessary correction factor, and documenting this data in a notebook.

6.3.3 Environmental Conditions

The assembled diffusion cell is placed in a constant temperature bath where the temperature is maintained at 25°C within 5°C. To prevent evaporation, be sure to periodically refill the temperature bath.

6.4 Control of Samples

6.4.1 Samples will be controlled according to QP-08.1.

6.5 Implementing Procedure

6.5.1 Preparation of cell assembly and rock slab

6.5.1.1 Fabricate the Plexiglas mount and the solution reservoirs of Plexiglas (see parts A and C of Attachment 1) using commercially available tools (e.g., lathe, mill, and/or drill press). The reservoirs may vary in size.

6.5.1.2 Manipulate a qualified specimen to fabricate a rock slab by diamond sawing the specimen to approximately the desired length (according to DP-60).

6.5.1.3 Cut the slab to the same thickness as the Plexiglas mount using an isomet polisher or diamond bit used on a lathe.

6.5.1.4 Seal the rock slab into the Plexiglas mount with Silastic (see part B of Attachment 1) and set the assembly aside to dry for approximately two weeks to cure the Silastic (adhesive chalking).

6.5.1.5 Photocopy both sides of the rock slab in the Plexiglas mount and determine the surface area of the photocopy image using a planimeter. If the slab is symmetrical, the area may be calculated. Record the data in a laboratory notebook.

6.5.1.6 Measure the dimensions of the rock slab using commercially available hand tools (micrometer or caliper) and record these dimensions in the laboratory notebook.

6.5.1.7 Ensure that the following data are recorded in a notebook:

- The qualified specimen ID number and a unique identifier assigned to the part of the rock slab utilized in this experiment.
- Dimensions of the rock slab
- Surface area of both sides of the rock slab and any necessary corrections to the planimeter or photocopy used.

6.5.2 Determination of Saturated Weight of Tuff

6.5.2.1 Determine the dry weight of a representative piece by drying an adjacent piece of the tuff core in a vacuum oven at 40-50°C until

a constant weight is obtained. Weigh the piece weekly until the weight stabilizes within 0.05 g and record the dry weight.

6.5.2.2 Determine the saturated (wet) weight of the piece by placing the piece in a small beaker of deionized water. Place the beaker in a vacuum oven at room temperature. Weigh the piece weekly until the weight stabilizes within 0.05 g and record the wet weight.

6.5.2.3 Measure the diameter and length of the representative piece and record the data in the notebook.

6.5.2.4 Ensure that the following data are recorded in the notebook:

- diameter and thickness of the representative piece of the rock slab specimen
- dry weight and wet weight in grams of the representative piece
- unique identifier of the balance used

6.5.3 Saturation of Rock Slab

6.5.3.1 Equilibrate the rock by placing the mounted rock slab in a beaker of the appropriate ground water (specified by the PI).

6.5.3.2 Place the beaker in a vacuum oven at room temperature.

6.5.3.3 Weigh weekly until a constant weight is obtained within 0.05 g.

6.5.3.4 Check the pH of the equilibrating water periodically according to DP-35. The pH should be in the range between pH 7 and pH 8.

6.5.3.5 If pH adjustment is needed, use dilute NaOH or HC1 (<0.1M) to make the adjustments.

6.5.3.6 Record the final pH of the solution after the rock slab is saturated.

6.5.4 Preparation of Tracer Solution

6.5.4.1 Prepare a solution of the appropriate ground water containing the tracer(s) to be used. The source of the ground water and traces(s) are specified by the PI. Record the type of tracer and the method of preparation in the notebook.

6.5.4.2 Measure the pH of the solution according tp DP-35 and record in the notebook.

6.5.4.3 Place an aliquot of the tracer solution in a capped container (the tracer concentration of this aliquot will be used to determine the initial concentration of the tracer).

6.5.5 Experiment

6.5.5.1 Assemble the mounted slab using O-rings and the bolts (as shown in Attachment 1) to hold the cell together.

6.5.5.2 Determine the volumes of the solution reservoirs within 2% by filling the reservoirs with clean water and pouring the water into a graduated cylinder, or by weighing the empty reservoirs then filling with water and weighing again. Plugs are utilized to seal the cell after the appropriate solutions have been places in parts A and C of the cell (see Attachment 1).

6.5.5.3 Fill the appropriate Plexiglas, reservoir with the tracer solution by placing a luer fitting in the threaded hole. Attach a 60 ml syringe barrel to the luer fitting, and pour the solution into the 60 ml syringe barrel until the reservoir is full. Fill the reservoir on the other side of the rock slab with the appropriate ground water. Plug the holes in the reservoirs. Place the cell in the constant temperature bath.

6.5.5.4 Record the start time (date and 24-hr clock time or Julian time).

6.5.5.5 Periodically sample the solution in the reservoir (at time intervals specified by the PI) that originally contained only ground water (the sample volume taken is usually 1 ml). Sampling is performed by removing the cell from the bath. Dry the cell with a kimwipe. Remove the plugs, and transfer an aliquot of the solution to be sampled to a vial while the same volume of ground water is replaced in the reservoir. Record the time at which the sample is taken and the volume of sample removed.

6.5.5.6 Analyze the concentration of tracer in the aliquot according to the appropriate analytical technique.

65.5.7 At the conclusion of the experiment (determined by the PI based on the analytical results of the periodic samples), determine the pH (according to DP-35) of the solution in the non-tracer reservoir and record the data in the notebook.

6.5.5.8 Ensure that the following entries are recorded in a laboratory notebook:

- final pH of the equilibrating water

- pH of the tracer solution method of preparation of the tracer solution
- volume of the reservoirs
- start time (date and 24-hr clock time or Julian time) at which the tracer solution and appropriate ground water were placed in the reservoirs
- amount of tracer solution aliquotted from the cell and transferred to the vial(s)
- date and 24-hr clock time or Julian time at which the sample(s) was taken
- analytical technique used or reference to the DP that was followed to determine the concentration of tracer in each sample
- pH of the solution after the experiment is finished

6.6 Data Acquisition and Reduction

The active recording of data as specified above will constitute the data acquisition. Computer programs such as word processing editors and spreadsheets will be used for recording and formatting data but are not part of the data acceptance criteria. Users should verify that the data has been recorded properly (e.g., saturated weight is greater than dry weight, etc.).

6.7 Deviations from the DP and Potential Sources of Error and Uncertainty

Any deviations from this procedure will be documented in the user's notebook. If the deviations are deemed critical by the PI, then a written statement will be put into the user's laboratory notebook evaluating the potential source of error and uncertainty.

7.0 RECORDS

Records generated as a result of this DP are entries in laboratory notebooks or attachments to laboratory notebooks. The documentation should consist of any applicable items identified in Section 6.0 of this procedure. Laboratory notebooks should be kept in accordance with QP-03.5.

All records should be submitted to the Records Processing Center in accordance with QP-17.6.

8.0 ACCEPTANCE CRITERIA

Proper recording of the data specified in Sections 6.5.1.7, 6.5.2.4, and 6.5.5.8 constitute the acceptance criteria for this DP. If no critical deviations (see Section 6.7) were made, these data will be accepted as qualified data for YMP.

9.0 TRAINING

9.1 Prior to conducting work described in Section 6.0, the user requires training to this procedure.

9.2 Training to this procedure is accomplished by “read only”. Training will be documented in accordance with QP-02.7.

10.0 ATTACHMENTS

Attachment 1: Schematic of cell assembly (1 page)

SCHEMATIC OF CELL ASSEMBLY